BULGUR WAFER AND ADJUNCTS FOR FALLOUT SHELTER RATIONS - OCD - WORK UNIT NO. 1311A • WRRL - ARS - USDA •

BULGUR WAFER AND ADJUNCT
FOR FALLOUT SHELTER RATIOI

A REPORT OF RESEARCH CONDUCTED JULY 1964 – JUNE 1965

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BULGUR WAFER AND ADJUNCTS

FOR FALLOUT SHELTER RATIONS

A report of research conducted July 1964 - June 1965

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BULGUR WAFER AND ADJUNCTS FOR FALLOUT SHELTER RATIONS

SUMMARY

Initial development work on the bulgur wheat wafer (the basic fallout shelter ration) and on adjuncts (foods used to modify or supplement the wafer) was reported in "Food Supply for Fallout Shelters" CDM-SR-60-62 Nov. 1962. Continuing and expanding work on wafer and adjunct development, modification, storage, and evaluation was detailed in the annual reports ("Bulgur Wafers and Adjuncts for Fallout Shelter Rations") for fiscal years 1962, 1963, and 1964. We now report a continuation of these efforts.

Shelf life (stability) may well be the single most important factor in determining the ultimate cost of a stockpiling program. Development of reliable surveillance testing methods to measure stability may depend upon an understanding of the mechanisms of deterioration. Major emphasis during FY 1965 has been in this area. Further elucidation of the products and mechanism of rancidification have been gained, principally through gas liquid chromatography (GLC) and rapid-scan mass spectrometry (MS). Thirty-one of the 40 peaks on gas liquid chromatograms of the volatile fraction of an autoxidized model compound, methyl linoleate, have been at least tentatively identified. They include several hydrocarbons, esters, acetals, alcohols, aldehydes, ketones, and dioxolanes not previously reported in the literature. The major components were pentenal, hexanal, amyl formate, methyl octonoate, and substituted dioxolanes. In addition, several complex higher-molecular-weight compounds have been identified by fractionation and GLC plus mass spectral analyses of the non-volatile residual oils of autoxidized methyl linoleate. Of particular interest were several alkyl-substituted trioxanes which smell rancid. Several of these compounds may be better indicators of incipient rancidity than hexanal which has been suggested before.

The consumption of oxygen by autoxidizing methyl linoleate, coupled with the appearance of low-molecular-weight aldehydes in the volatile fraction, tends to confirm that hydroperoxides undergo cleavage to products which in turn oxidize to low-molecular-weight aldehydes. Uptake of oxygen by methyl linoleate in four weeks was twice the theoretical amount required to form the monohydroperoxide.

The early appearance of the permanent gases--carbon monoxide, carbon dioxide, and hydrogen--in the vapors from autoxidizing methyl linoleate and their detection in headspace gases of bulgur and puffed bulgur stored under oxygen suggest the use of these fixed gases as indicators of incipient rancidity.

GLC techniques are now being applied to bulgur and puffed bulgur stored under high oxygen tension at elevated temperatures. We hope to develop a reliable, accelerated storage stability test. Thus far we have followed the development of hydrocarbons and carbonyls only. As in the model system, the carbonyl concentrations first increase and then decrease in storage, but the hydrocarbons continue to increase after their first appearance.

Work has continued on two alternate methods of processing the cereal ingredient: gun puffing and hot-air puff-drying. Gun puffing of both wheat and bulgur can be manipulated to produce wide ranges in the degree of expansion and in the texture of the kernels. Gun-puffed bulgur has a better texture and flavor than gun-puffed wheat. In hot-air puff-drying, wheat is soaked for up to 3 days and then puffed in hot air without intervening steaming. With proper precautions to prevent undesirable microbial growth, the product has excellent and somewhat different flavor, high puff indices (2.5-3.0), and good texture (200-250 m-g torque). Perhaps enzymatic or microbial action during soaking develops these desirable characteristics. Improvement of both texture and flavor by controlled enzymatic or microbial action appears promising and worth further investigation.

The final phase of adjunct development was preparation of a high-methoxyl jelly mix that can be reconstituted in cold water. Comments from two Civil Defense sources were almost completely favorable.

Until an objective accelerated test for stability is developed and proven, subjective examination by test panels of products stored at normal temperature remains the one sure measure of shelf life. Such evaluation of both wafers and selected adjuncts is continuing under a five-year contract with Oregon State University. After 28 months' storage of wafers under a variety of conditions, panel results strongly confirmed the protective action of packaging in nitrogen. The protective action of malt syrup in the recipe is also becoming more significant. Chemical tests on the storage samples show

significant changes in nearly all properties being checked. These changes depend to varying degrees on the main storage variables, especially temperature. When the storage tests near completion, correlations between panel evaluations and the chemical and physical measurements may uncover a reliable objective method for evaluating stability.

Results of panel evaluations after 18 months' storage of the twelve selected adjuncts indicate a protective action by nitrogen packaging and a secondary protective action by desiccants. Analysis of headspace gases from samples stored at 100° showed that carbon dioxide develops rapidly if the samples contain amino acids or peptides. A Maillard-type browning reaction may be taking place.

BULGUR WAFER AND ADJUNCTS FOR FALLOUT SHELTER RATIONS

SECTION 1

DEVELOPMENT OF NEW STABILITY-EVALUATION METHODS

In order to establish reliable objective measurements that can be applied in surveillance testing of stored food products, such as the bulgur wafer, it is necessary to understand the chemistry of their deterioration and its relation to development of off-flavors or aromas. We stated before that the component in bulgur shelter wafers most likely to limit their shelf life is the natural wheat lipids. That premise continues to guide our research. No reason for changing it has appeared.

For simplicity much of the work has been performed on a model system, methyl linoleate. Linoleic acid makes up over half of the fatty acids found in wheat lipids and is one of the more unstable. However, we have also done limited work on bulgur in various forms and under various conditions of accelerated storage to provide means for evaluating changes in formulation and processing.

A Model System - Methyl Linoleate

Work continued on rigorous identification of the volatile substances generated in autoxidizing methyl linoleate. We used principally capillary gas liquid chromatography (GLC) coupled with rapid-scan mass spectrometry (MS), plus preparative GLC, infrared spectroscopy (IR), and nuclear magnetic resonance (NMR). In addition we began research on identification of some high-boiling components from the residual oil of oxidized methyl linoleate by the techniques previously mentioned, plus some classical chemical procedures.

In preliminary work, electron paramagnetic resonance revealed free radicals in methyl linoleate under nitrogen atmosphere and at the temperature of liquid nitrogen and when exposed to ultraviolet light. This work has not been extended during this period because of difficulty in preparing suitable model compounds to assist in the interpretation of the data.

Volatile components

Vapors from the autoxidizing methyl linoleate were collected by passing oxygen at the rate of 10 ml/min through methyl linoleate on purified glass wool, all at room temperature, and passing the exit gases through a U-tube trap at -78°C to condense out the volatiles. The condensate was then extracted with 2,2,4-trimethyl pentane, and

the extract analyzed on a 200-ft x 0.01-in. capillary column coated with General Electric Silicone Oil SF-96 (50). The temperature-programmed chromatogram obtained with a flame ionization detector contained 40 fairly prominent and well defined peaks. The chromatogram in Figure 1.1 is typical of several replicate runs.

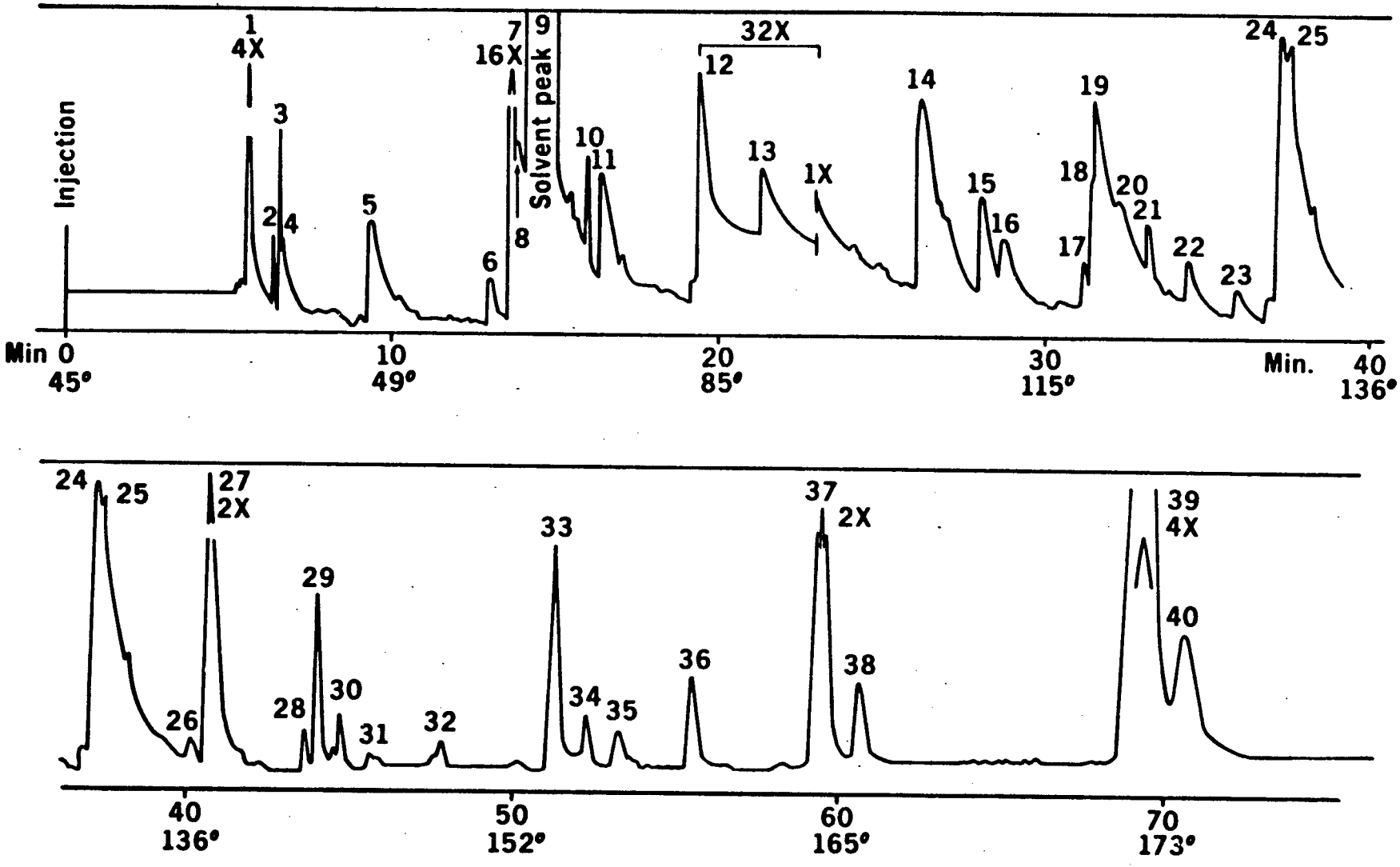
Minor differences sometimes occurred in the first 4 peaks in replicate runs. For example, propanol (peak 4) was definitely present in measurable quantities in the chromatogram from one oxidation experiment, but in a second oxidation it was barely detectable. In another instance, 1-pentane (peak 2) was a substantial peak, whereas in most runs it was barely detectable. Beyond peak 4, however, GLC chromatograms from many replicate oxidations lacked significant differences.

For corroborative mass spectral analysis, the effluent end of the GLC column (minus the detector) was connected directly to the vacuum manifold leading to the ion chamber of a Bendix time-of-flight mass spectrometer (rapid-scan mass spectrometer) (Figure 1.2). Conditions were adjusted so that components took the same length of time to travel the column as in normal GLC runs. As each component emerged from the column, changes in the mass spectrum were observed on an oscilloscope, and the complete mass spectrum was simultaneously recorded on a Minneapolis-Honeywell Visacorder. A mass range of 20-200 mass units was scanned in 1-3 seconds. As with GLC chromatograms, no significant differences in replicate oxidations were observed beyond peak 4

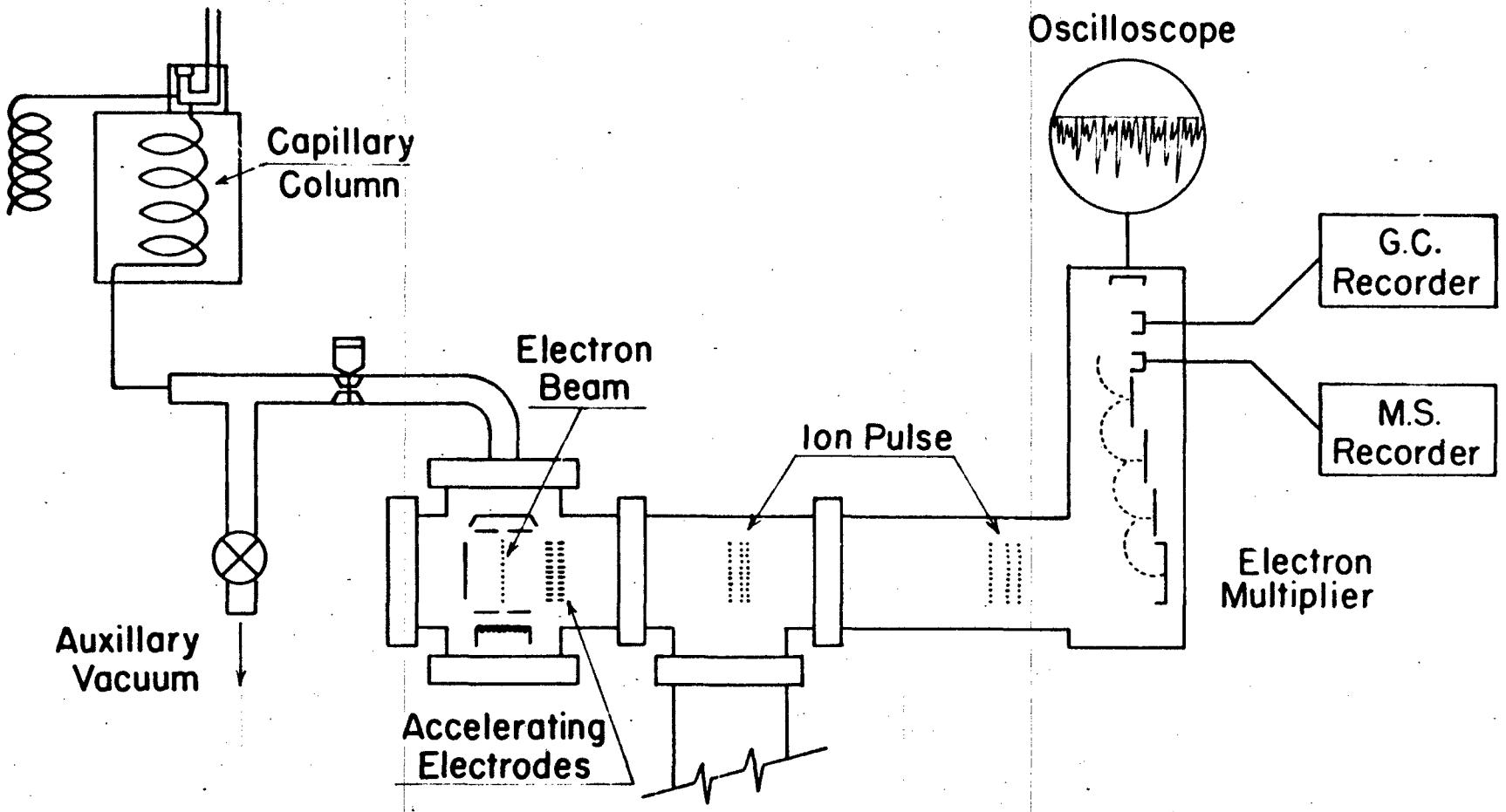
The compounds identified by these methods appear in Table 1.1. Except where noted, the mass spectral data were compared with those of authentic compounds or with literature data. In addition, most compounds were confirmed by comparison of GLC retention times. The list includes several hydrocarbons, esters, acetals, alcohols, aldehydes, ketones, and dioxolanes not previously reported in the literature of lipid oxidation. The major components were pentanal, hexanal, amyl formate, methyl octanoate, and substituted dioxolanes.

The oxidizing technique employed in this study is very mild and probably represents well the conditions in actual storage of the wafer. The mild oxidation may account for the large number of compounds which we identified, but which have not been identified by other workers who used more vigorous oxidizing conditions in similar studies. The mildness may also account for the absence of acetaldehyde, heptenal, 2-octenal, 2-nonenal, 2,4-nonadienal, and 2,4-decadienal commonly reported by other workers in the field.

The acetals and dioxolanes in Table 1.1 suggest that some of the simple carbonyls reported by others, but not found in these studies, may be present in the nonvolatile fraction as higher molecular weight complexes.



• Figure 1.1 • Gas chromatogram of volatile oxidation products.



• Figure 1.2 •

Schematic diagram of capillary chromatograph and time-of-flight mass spectrometer.

TABLE 1.1.--Volatile compounds from autoxidation of methyl linoleate

Peak[a] No.	Mass spec. and GLC confirm.	Mass spec. identification alone
1	methyl formate	
2		1-pentene[b]
3	n-pentane	
4	propanal	
5	n-butanal	
6		2-pentanone[c]
7	n-pentanal	
8		no identification
9	isooctane-solvent	
10	solvent impurity	
11	n-butanol	
12	n-hexanal	
13	n-amyl formate	
14	2-heptanone	
15	methyl hexanoate	
16	n-hexanol	
17	1,1-dimethoxy-n-hexane	
18	(2-heptenal)	
19	(1-octene-3-one)	
20		no identification
21		" "
22	methyl heptanoate	
23		no identification
24		" "
25		" "
26		" "
27	methyl octanoate	
28		no identification
29		2-ethyl-4-pentyldioxolane
30		isomer of 29
31		no identification
32		" "
33		2-propyl-4-pentyldioxolane
34		isomer of 33
35	n-pentyl-n-hexanoate	
36	1-methoxy-1-n-pentoxy-n-hexane	
37		n-butyl-4-pentyldioxolane
38		isomer of 37
39	2,4-dipentyldioxolane	
40	isomer of 39	

[a] Refer to chromatogra_ of Fig. 1.1. Some small peaks have not been numbered.

[b] Suggested but not confirmed.

[c] Good mass spectral evidence. Retention time off by 15 sec.

Non-volatile residual fraction

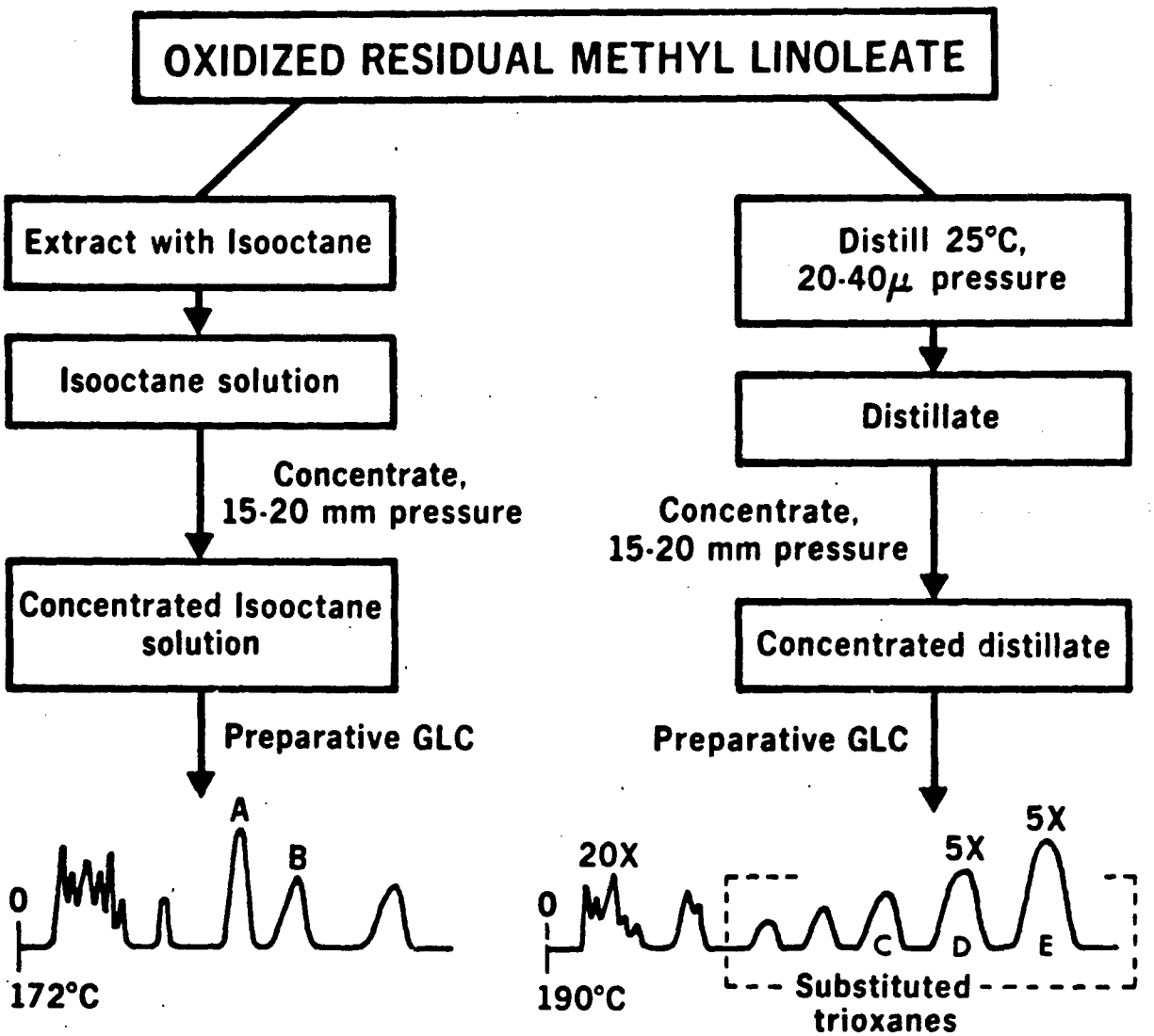
The non-volatile residue from autoxidized methyl linoleate also may contain degradation products important in the overall sensory perception of deterioration. We have used two approaches to isolate and identify the decomposition products (Figure 1.3). In both pathways a 4-1/2-ft. x 1/4-in. stainless-steel preparative GLC column containing 20% Apiezon M on Chromosorb P was used for the final separation.

Several components were isolated in the extraction procedure. The two most prominent ones (A & B, Fig. 1.3) have been rigorously identified by MS, IR and NMR.

To confirm the identity of compound A, we synthesized an authentic sample of 8-formyl methyl octanoate by a preparative scheme from the literature. The IR and mass spectra of the authentic sample of 8-formyl methyl octanoate were identical to those of compound A. Compound B was identified by comparison of its IR and mass spectra with those of two other members of the same homologous series, 9-formoxy methyl nonanoate and 10-formoxy methyl decanoate, as well as 10-formoxy ethyl decanoate.

A second fraction of the residue from autoxidized methyl linoleate (peroxide number of approximately 1000) was distilled and concentrated. The final concentrate smelled like rancid food.

We isolated five components from the concentrated distillate, using the preparative GLC Apiezon column at 190°C. The IR spectra of these five compounds were very similar and showed the ether linkage as the only functional group. The most abundant member of the series (E) was hydrolyzed in 2 M aqueous methanolic hydrochloric acid, and the resulting carbonyl compounds were converted into 2,4-dinitrophenyl-hydrazones. Paper chromatography of the hydrazones revealed hexanal and a high-molecular-weight carbonyl compound. A second portion was hydrolyzed and the carbonyls isolated from the reaction mixture with ether and separated in a 6-ft. GLC column containing 15% Carbowax 20 M on Chromosorb P. The mass spectra of these two fractions showed they were hexanal and a C_{12} unsaturated carbonyl. It seems probable that hexanal was the principle hydrolysis product, and the C_{12} unsaturated carbonyl was an aldol condensation product which formed from hexanal during hydrolysis. Consequently this series of compounds is probably trioxanes. To confirm this possibility, 2,4,6-tripentyl trioxane was synthesized from hexanal, purified by preparative GLC, and analyzed by MS. Component E was shown to be 2,4,6-tripentyl trioxane, since its MS and IR spectrum were identical with those of the authentic samples. In further comparisons, 2-butyl-4,6-dipentyl trioxane and 2,4-dibutyl-6-pentyl trioxane were synthesized and shown to be identical by MS and IR with two other members of the homologous series (C & D respectively). Work is in progress to isolate enough of the remaining two members of the series for MS analysis.



•Figure 1.3 • Scheme for isolation of components from oxidized residual oil.

The isolation and identification of these complex higher-molecular-weight compounds from the non-volatile residue further illustrates the complexity of fat oxidation and the need for basic studies of pure model systems such as methyl linoleate. The alkyl-substituted trioxanes are particularly interesting because they smell rancid. Their appearance and increase may more closely parallel the development of sensory rancidity in a stored product than other indicators. This possibility will be explored in continuing work.

Oxygen uptake

The rate of oxygen uptake by methyl linoleate suspended on purified glass-wool at room temperature in an atmosphere of oxygen under laboratory light has been studied to obtain more information about the cause of development of rancidity in the ester. Four-gram samples of methyl linoleate (97 percent purity) were placed on purified glass-wool in two 100-ml flasks, each fitted with a rubber septum. The flasks were then connected to a gas burette equipped with a mercury leveling bulb. The gas in the system was replaced with oxygen purified by passing it through a stainless-steel tube containing copper oxide at 350° and then through tubes containing magnesium perchlorate (anhydrone) and asbestos coated with sodium hydroxide. Analysis of the oxygen by gas chromatography revealed no carbon monoxide, carbon dioxide, or hydrogen. Oxygen uptake was followed over four weeks in one flask; the second was the source for GLC detection of the development of permanent gases, as is described below.

The rate of oxygen uptake is an S-shaped curve with time. Uptake is slow during the first seven days, very rapid for the next ten days, and decreases slowly thereafter. The methyl linoleate consumed twice the oxygen theoretically required to form the monohydroperoxide. Day (Lillard, D. A. and Day, E. A., 1964. J. Am. Oil Chemists' Soc. 47: 549) has shown that unsaturated aldehydes can be oxidized by oxygen at room temperature to several lower-molecular-weight aldehydes. Such oxidation may account for the high oxygen uptake and the appearance of low-molecular-weight aldehydes in our model system.

Permanent gases

It is well established that hydroperoxides form during oxidation of methyl linoleate, and various workers have shown that thermal decomposition of primary hydroperoxides produces the gases, hydrogen, carbon monoxide, and carbon dioxide. We suspected that these fixed gases would form during the early stages of oxidation of the model system and that their appearance might be a good test for incipient rancidification in bulgur and similar products. Analysis for carbon monoxide and carbon dioxide in headspace vapors over stored products would be much simpler and would require less elaborate equipment than would analysis for hydrocarbon or carbonyl.

To find fixed gases, vapor samples were withdrawn periodically from the second of the two flasks described in the preceding oxygen uptake section.

Carbon dioxide analysis was on a 16-ft. by 1/4-in. o.d. stainless-steel column containing 33 percent hexamethyl phosphoramide (HMPA) on firebrick and with a helium exit flow rate of 30 ml. per minute and a column temperature of 22°C. Analysis of carbon monoxide was on a 12-ft. by 1/8-in. o.d. stainless-steel column containing Linde molecular sieve Type 5A and with a helium exit flow rate of 30 ml. per minute and a column temperature of 50°C.

Analysis for hydrogen was made on a Fisher Gas Analyzer, Model 25V, equipped with a 6-ft. by 1/8-in. o.d. stainless-steel column containing HMPA on firebrick, followed by a 12-ft. by 1/8-in. o.d. stainless-steel column containing Fisher Type 13A molecular sieve and with an argon exit flow rate of 80 ml. per minute and a column temperature of 22°C.

Carbon monoxide, carbon dioxide, and hydrogen were first detected in the samples after seven days. At that time the methyl linoleate had consumed approximately one-third of the theoretical amount of oxygen required to form the monohydroperoxides. We continued to analyze vapor samples from the autoxidizing ester for the next 4 weeks; all fixed gases increased in concentration with time.

In order to eliminate the possibility that the observed fixed gases were artifacts, we ran blanks with each GLC analysis. The blanks were prepared by drawing the appropriate volume of oxygen, several-fold larger than that used for permanent gas analysis, from a parallel apparatus containing only purified glass wool and injecting it into three different GLC chromatographs. Chromatograms from the blanks showed no peaks with retention times corresponding to those of authentic samples of carbon monoxide, carbon dioxide and hydrogen. These gases, perhaps, offer a good possibility for development of a rapid, objective test for incipient rancidity of the stored wafers or other fat-containing foods.

Components of Bulgur Vapors

Work has continued with GLC to identify the volatiles from autoxidizing bulgur. We plan now to use the analytical techniques developed for our model system to analyze completely the vapors of rancid bulgur.

Previous work (Bulgur Wafer and Adjuncts for Fallout Shelter Rations, 1964) showed the presence of saturated hydrocarbons and carbonyls, and the present work shows carbon monoxide and carbon dioxide in head-space gases over ground bulgur and puffed ground bulgur.

The analyses for carbon monoxide, carbon dioxide, and hydrogen were by the techniques described for permanent gases in the model system. These analyses were carried out on samples of headspace gas over ground bulgur and ground puffed bulgur that had been stored under oxygen at 70° and 90°F for 17 months.

Analyzing for hydrogen in these headspace samples was difficult because hydrogen, if present, occurs in such minute quantities. GLC chromatograms from headspace gases of ground rancidifying bulgur under oxygen did not consistently show the small peak with a retention time corresponding to that of hydrogen. The presence of carbon monoxide and carbon dioxide, however, was well established.

Acceleration of Rancidity

Attempts to devise an accelerated storage test to estimate storage stability of bulgur wafers are continuing. In our model system, methyl linoleate, hydrocarbons emerged before off-flavor developed or before the accompanying carbonyl compounds appeared. We are trying to determine whether the same sequence holds in bulgur and products containing bulgur. If hydrocarbons precede off-flavor development in bulgur, they might be an exceedingly sensitive indicator of incipient rancidity.

Freshly prepared ground bulgur and ground hot-air-puffed bulgur were sealed under oxygen in tin cans and stored at 34°, 70°, 90°, and 100°F. Controls were samples sealed under nitrogen and stored at the same temperatures. We took vapor samples (1 to 3 ml) from the cans and analyzed them by GLC for hydrocarbons and carbonyls, principally hexanal. Shortage of time and equipment precluded analyses for permanent gases in this series of experiments. After vapor sampling, the materials were checked by sensory methods for development of rancid flavor or aroma.

Headspace samples from ground puffed bulgur stored under oxygen at 90° and 100°F showed an increase in hexanal and pentanal after two weeks, but five weeks passed before pentane began to increase. During longer storage at 100°F, hexanal and pentanal decreased, but pentane continued to increase. Hexanal and pentanal arose similarly from ground unpuffed bulgur but over longer periods. Under nitrogen, neither product showed any significant changes in the levels of pentane, hexanal, or pentanal. The taste panel judged none of the samples rancid through 18 weeks of storage.

To analyze both classes of compounds simultaneously, two parallel pieces of chromatographic equipment were used. The one used for carbonyls was sensitive to 10 times less material than the one used for hydrocarbons. This difference in sensitivity may account for

the detection of carbonyls before hydrocarbons from the puffed bulgur although the order was the reverse from the model system. In any case, the significant observation is that concentration of pentane increases with time, whereas the carbonyls reach a peak of concentration and then decline.

Studies with the model system, methyl linoleate, continue to shed light on the mechanism of oxidative deterioration leading to rancidity. The discovery of substituted dioxolanes, not previously reported, in the volatile fraction, provides evidence of an alternative pathway of oxidation: The 11-hydroperoxide of methyl linoleate could cleave to form a reactive pentyl ketene type of compound which, in turn, could react with the aldehydes formed. We have tentatively established the formation of the 11-hydroperoxide.

A second series of cyclic ethers, the trialkyl trioxanes appear in the non-volatile residue of oxidized methyl linoleate. They may form from aldehydes or some free radical aldehyde precursor. The most common of the series, 2,4,6-tripentyl trioxane, is a trimer of hexanal, the principal aldehyde formed in early stages of oxidation.

The consumption of aldehydes in these reactions could explain the discrepancy between the formation of aldehydes (especially hexanal) and the slow development of rancid taste or aroma.

The work underway with bulgur and puffed bulgur under high oxygen tension and high temperature suggests that the lipids in these systems follow the same general reactions and pathways as those in the model system. This, of course, needs much confirmation with more extensive and exhaustive studies.

SECTION 2

ALTERATIONS IN PROCESSING OF CEREAL INGREDIENT

We are still trying to improve the wheat in the wafer through changes in its texture and flavor. We have considered lowering the cost by using raw wheat or simplifying the process of expanding the wheat material. A new hot-air puffer, which simulates the commercial puffer at Van Brode Milling Company, has been put into operation and used to delineate the effects of major operational variables.

Gun Puffing

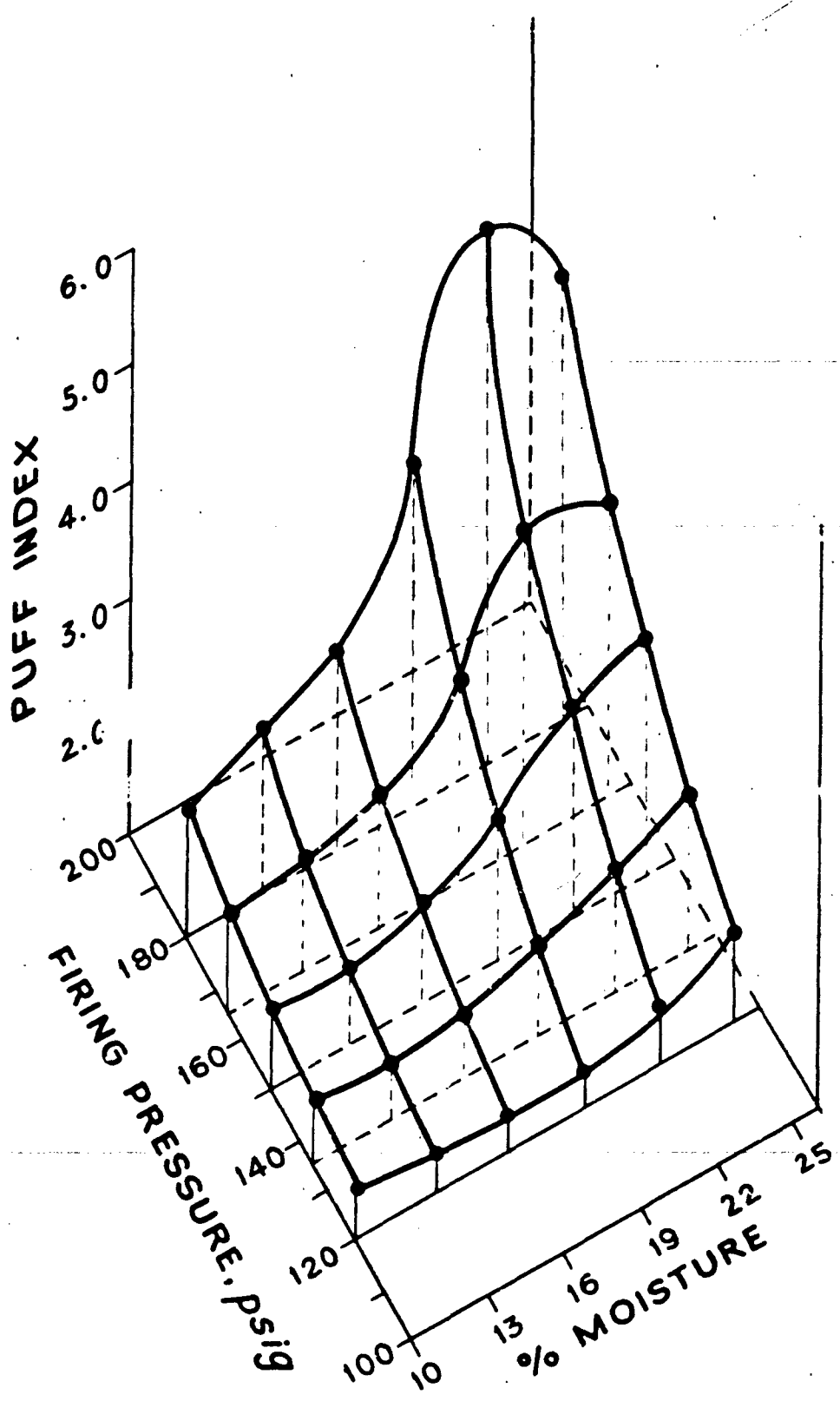
The study of gun-puffing of wheat ("Bulgur Wafers and Adjuncts for Fallout Shelter Rations," 1964) has now been extended to bulgur to see if the texture and flavor of gun-puffed bulgur are superior enough to justify the slightly greater expense.

A single lot of whole-kernel bulgur, commercially prepared from hard red winter wheat by an atmospheric cooking process, was puffed. Portions were adjusted to 10, 13, 16, 19, 22, and 25% moisture, and samples of each moisture level were fired from the gun at pressures of 120, 135, 150, 165, and 190 psig. The puffed product was then evaluated by measuring (a) puff index (bulk density), (b) texture, on the Brabender Hardness Tester, (c) soluble starch, and (d) color or amount of toasting.

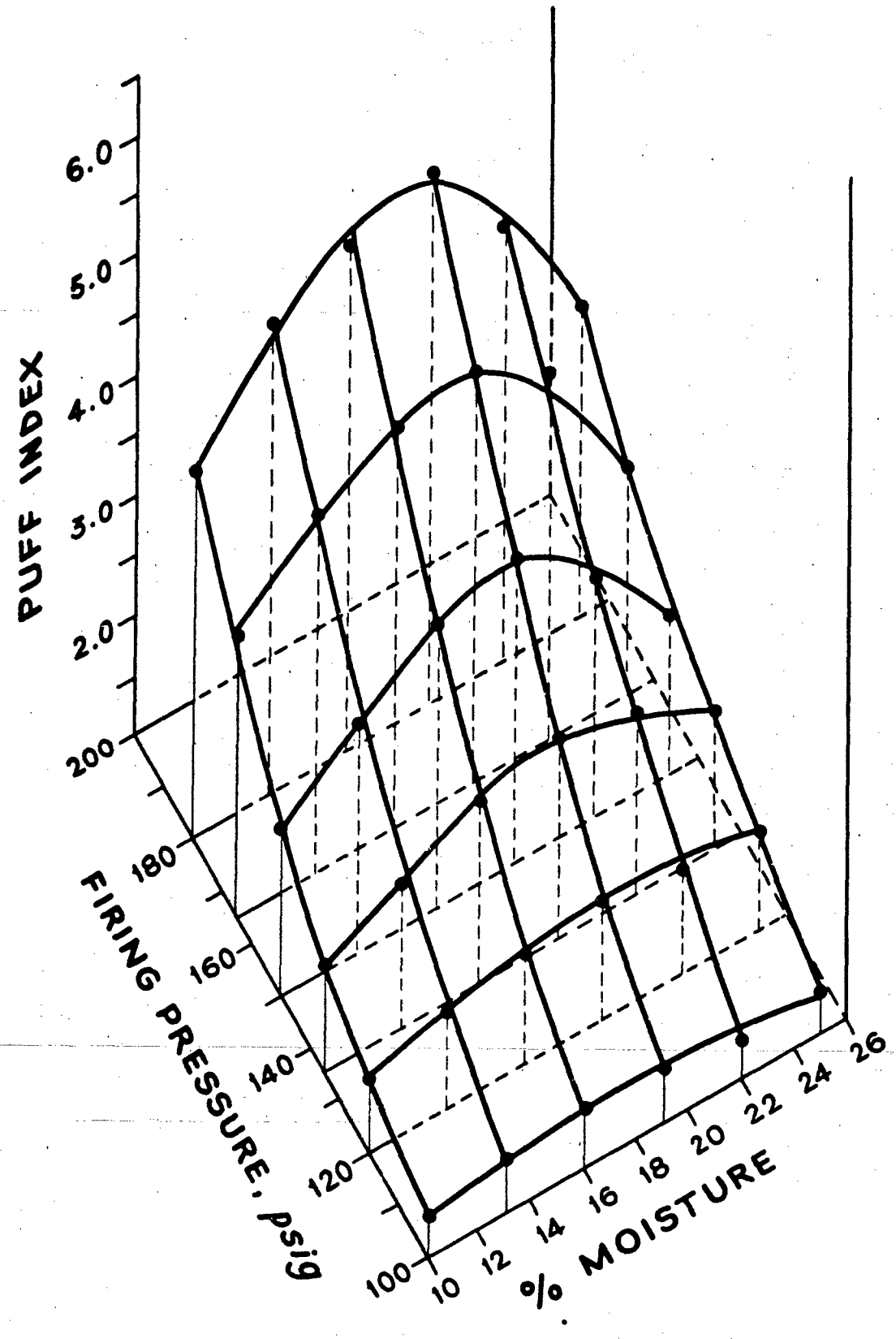
Moisture appears to have a much greater effect on puff index of bulgur than it does on wheat, as can be seen by comparison of Figures 2.1 and 2.2. Only at the higher moistures, 19% and above, does firing pressure begin to show a marked effect on bulgur. Whether 22% is in fact the optimum moisture for expansion, as indicated in Figure 2.1, is not entirely clear. The plastic moist puffed bulgur tends to recompress or deform when it hits the surfaces of the receiving hopper. This deformation is readily visible in the 25% moisture samples fired at the higher pressures.

Textural values are related to puff index (Figures 2.3 and 2.4), but bulgur differs from wheat in that there is apparently no moisture dependence as indicated in the wheat. In addition, bulgur is appreciably more tender than wheat at all degrees of expansion, except the very lowest (<1.5).

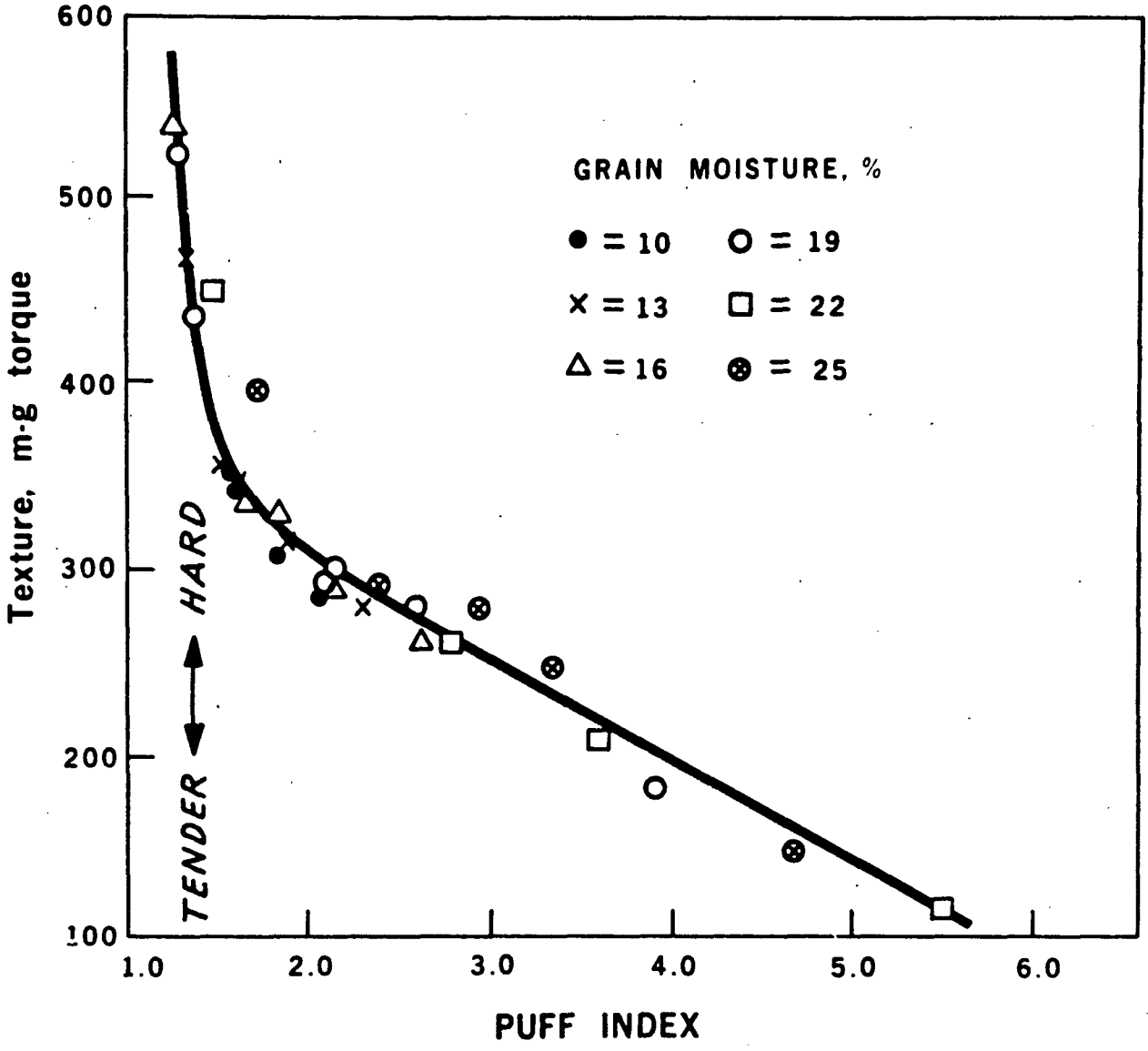
At 16% moisture and below, gun-puffed wheat showed one rate of starch solubilization over the range of puff index, but the rate increased as moisture rose from 19% to 25% (Figure 2.5). Perhaps starch at 16% moisture and below is solubilized only by mechanical rupture of the starch granules, but at 19% and above more cooking (gelatinization)



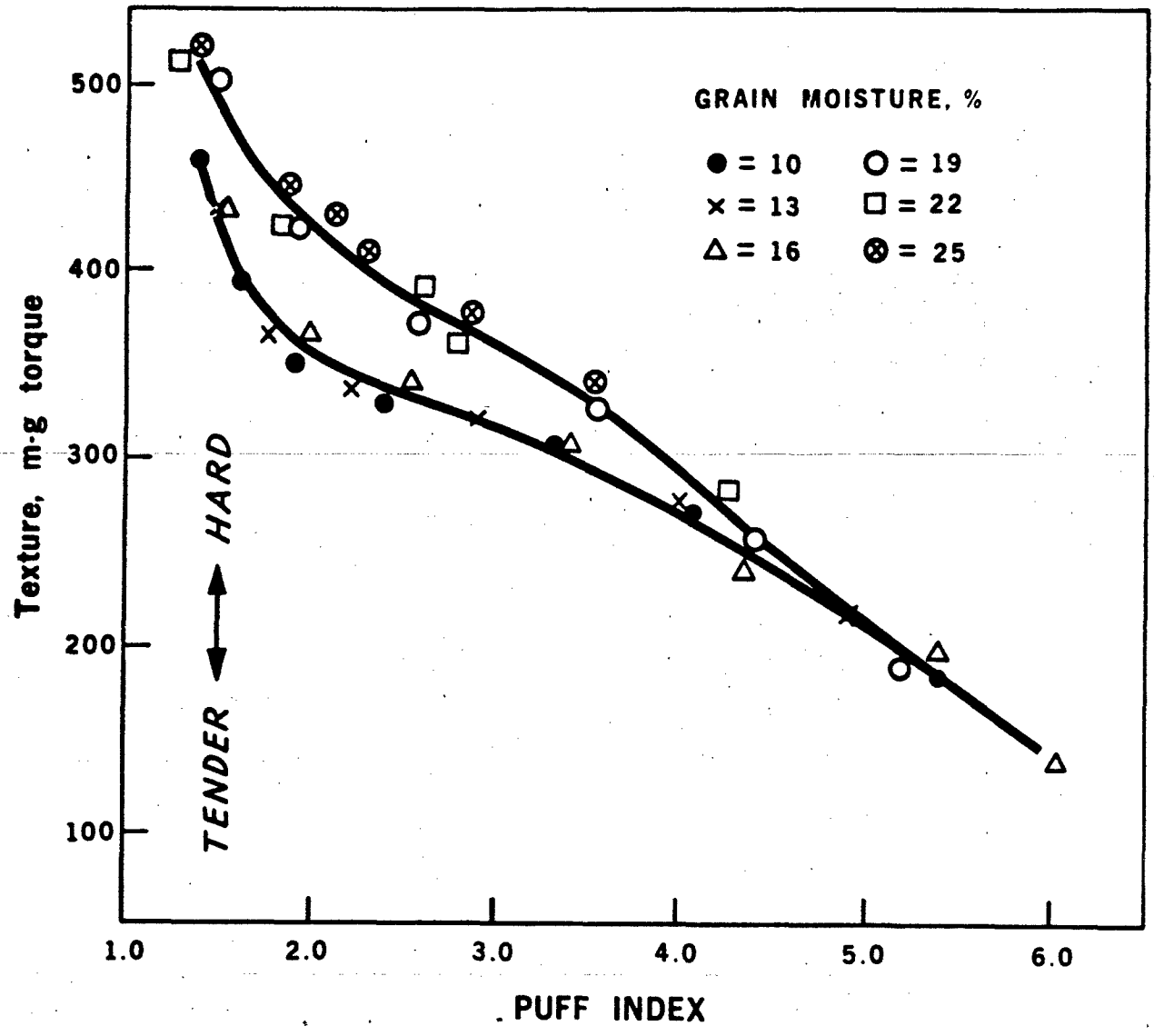
• Figure 2.1 • Relationship of firing pressure, moisture, and puff index in gun-puffed bulgur.



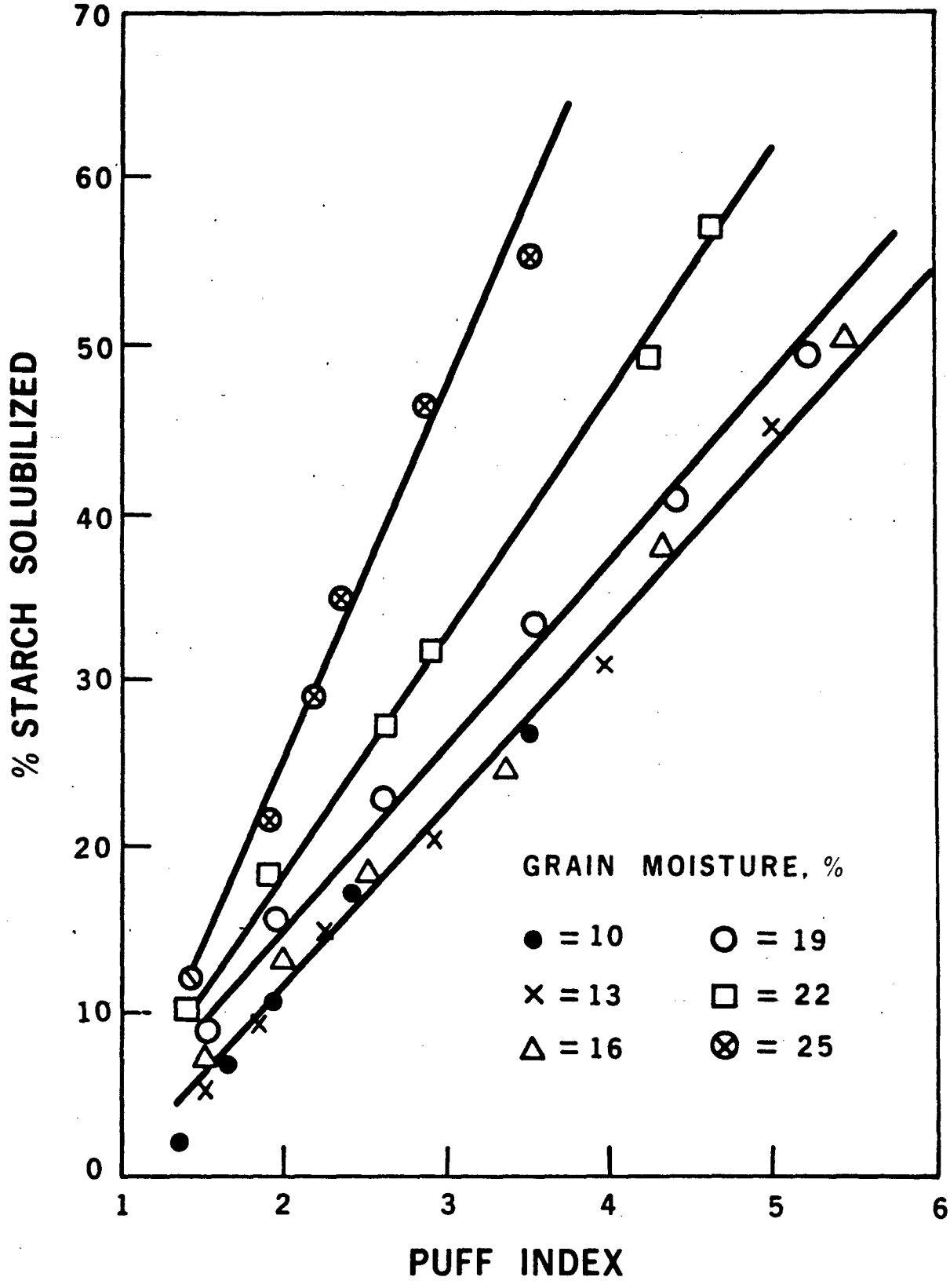
• Figure 2.2 • Relationship of firing pressure, moisture, and puff index in gun-puffed wheat.



• Figure 2.3 • Relationship of puff index, moisture, and texture in gun-puffed bulgur.



• Figure 2.4 • Relationship of puff index, moisture, and texture in gun-puffed wheat.



• Figure 2.5 • Relationship of puff index, moisture, and starch solubilization in gun-puffed wheat.

in the gun before explosive decompression may solubilize starch both by hydration and mechanical rupture of the granules. Or mechanical rupture may increase because of hydration effects. Gun-puffed bulgur, in contrast, shows that the rate of apparent starch solubilization decreases constantly as moisture increases (Figure 2.6). This suggests that amylose degrades in bulgur, perhaps hydrolytically, under the conditions of gun puffing or, less probably, the conditions in the gun strengthen the granule walls with the result that mechanical rupture on explosive decompression is less.

Color developed in the puffed bulgur about the same as it did in wheat. Overall darkness and redness of the samples increased with firing pressure. Slightly more toasting was evident in the low-expansion samples, probably because slight dextrinization of starch during gelatinization allowed additional caramelization or browning.

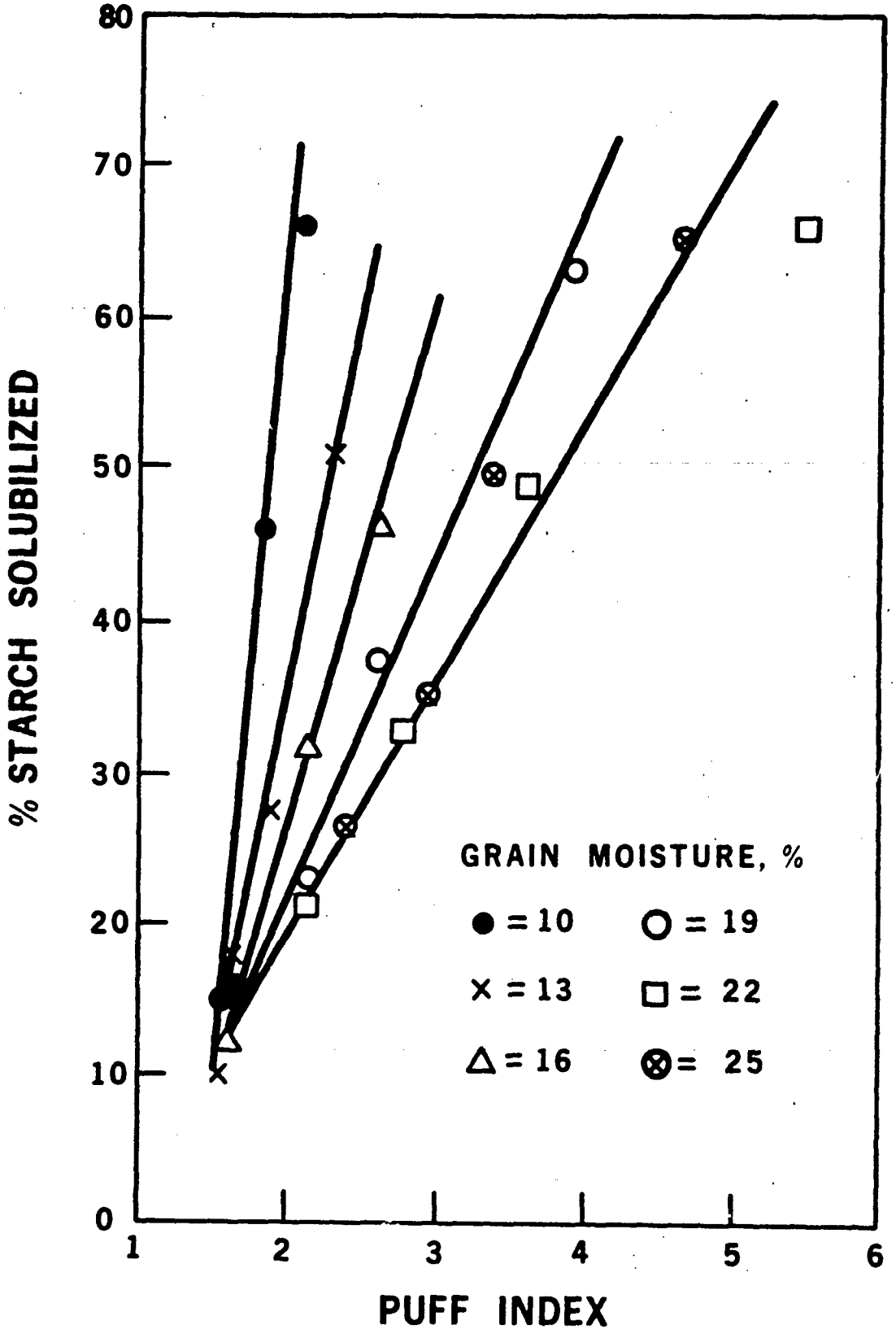
Gun-puffing offers the advantage over hot-air puffing of providing ingredients with expansions greater than 2-fold and consequently potentially more tender products. Several breakfast cereal companies already have such equipment in regular use.

Hot-Air Puff-Drying

An interesting modification of the hot-air puff-drying method of preparing the wheat ingredient was mentioned very briefly in our previous report ("Bulgur Wafer and Adjuncts for Fallout Shelter Rations," 1964). This technique has been investigated further.

When wheat is soaked in water at room temperature for two to three days, drained, and then exposed to hot air, it expands, apparently fully cooked and with good texture. Microbial action appears after the first day, and the final puffed product smells and tastes bad if this action is not controlled. Addition of 100-150 ppm available chlorine (as sodium hypochlorite solution) to the soak water effectively retards microbial action without noticeably affecting taste. Concentrations of chlorine above this level adversely affect flavor.

To investigate this process further, we soaked samples of hard red winter wheat for several time periods at several temperatures. After the soak, the samples were drained, the moisture content of the soaked wheat determined, and the samples puffed in hot air at 500°F with a velocity of 1800-2000 ft/minute. At a wet loading rate of 1 to 1-1/2 lb/ft^2, exposure for 70 to 90 seconds produced a lightly toasted, well dried product. Puff indices and hardness (Brabender Hardness Tester) of the puffed product are shown in Table 2.1



• Figure 2.6 • Relationship of puff index, moisture, and starch solubilization in gun-puffed bulgur.

TABLE 2.1.--Some characteristics of soaked hot-air-puffed wheat.

Soaking time, hr	Soaking temperature														
	Room temperature			100°F			130°F			150°F			170°F		
	Moisture, %	Puff index	Hardness, m-g	Moisture, %	Puff index	Hardness, m-g	Moisture, %	Puff index	Hardness, m-g	Moisture, %	Puff index	Hardness, m-g	Moisture, %	Puff index	Hardness, m-g
4	--	--	--	--	--	--	--	--	--	--	--	--	57	1.9	346
8	39	2.0	370	42	1.9	330	47	2.2	252	53	2.3	282	65	2.2	312
16	--	--	--	--	--	--	50	2.7	242	57	2.5	284	69	2.0	294
24	43	2.0	338	50	2.7	216	50	2.9	230	60	2.7	278	--	--	--
32	--	--	--	--	--	--	50	3.0	222	--	--	--	--	--	--
48	45	2.3	246	51	2.7	212	51	2.9	218	62	2.9	270	--	--	--
70	48	2.7	220	54	3.2	193	50	2.8	223	--	--	--	--	--	--

Either activity of the natural wheat enzymes or microbial activity, or both, probably make the wheat kernel ready for puffing. Temperatures of 100° and 130°F, which enhance such activities, generally provide products with maximum puff indices and minimum hardnesses. The soaked wet wheat which yielded puffed products with low hardness and high puff indices had a soft endosperm with consistency like toothpaste.

Soaked and hot-air puff-dried wheat resembles puffed bulgur in many ways and might be substituted for bulgur in wafers and other products. The fiber content of the product from whole wheat would be higher than in puffed bulgur. However, the fiber seems less noticeable and may not impair its usefulness. We produced a product lower in fiber, by first partially debranning the wheat in a pearler. Several degrees of debranning were tried. We found that the amount of debranning must be limited, perhaps to a level similar to that used for bulgur. Greater debranning exposed the endosperm in places; some kernels lost their shape during soaking; and stuck together during puff-drying.

The opportunity to modify both flavor and texture of expanded products by controlled enzymatic or microbiological action appears very promising.

Hot-Air Puffing and Textural Measurement

A new pilot model of the continuous hot-air puffer manufactured by the Surface Combustion Company has been installed. Puffing can be varied over a much broader range with this unit than was possible with the small unit formerly in use at this Laboratory. An investigation of the effect of altering these variables has been conducted. The parameters checked in the puffer were temperature (450°, 500°, and 550°F), air volocity (950, 1650, and 2200 fpm nominal), bed loading (1/2, 1, and 2 lb/ft^2), and residence time (15, 25, and 35 seconds). Two single lots of bulgur, one a red wheat bulgur from Farmers Cooperative Commission Co. and the other a white wheat bulgur from Armeno Cereal Co., were compared. Although results varied somewhat for the two lots (Tables 2.2 and 2.3) several generalizations can be drawn. The lowest air velocity (950 fpm) is inadequate to fluidize the grain bed except at 1/2 lb/ft^2 loading. Puff index generally increases with increasing air temperature and velocity, but control of maximum puffing without overtoasting becomes more difficult at the more severe conditions. Increasing residence time also brings about slight increases in puff index, but sometimes leads to overtoasting.

Textural values as measured on the Brabender Hardness Tester show consistent trends within each series of samples. Texture appears to be primarily a function of puff index; samples become more tender as puff index increases. There is evidence in both series, however, that increased residence time in the puffer, beyond that necessary for achieving maximum puff index, does make the product slightly more tender.

TABLE 2.2.--Effect of process variables on product characteristics - red wheat bulgur

Air temp. °F	Tray loading lb/ft²	Air velocity, f.p.m. [a] 965 — Residence time, seconds 15 PI [b]	15 H [c]	25 PI	25 H	35 PI	35 H	1650 — Residence time, seconds 15 PI	15 H	25 PI	25 H	35 PI	35 H	2200 — Residence time, seconds 15 PI	15 H	25 PI	25 H	35 PI	35 H
450	1/2	1.4	426	1.5	380	1.5	368	1.5	377	1.5	363	1.5	371	1.5	383	1.5	381	1.5	370
	1	1.3*	588	1.3	446	1.5	405	1.5	396	1.5	374	1.5	372	1.5	387	1.5	386	1.5	385
	2	-	-	-	-	-	-	1.4	480	1.5	391	1.5	391	1.3	461	1.5	393	1.5	386
500	1/2	1.5*	377	1.6*	271	1.6‡*	364	1.6	367	1.6‡	371	-	-	1.6	363	1.6‡	364	-	-
	1	1.3*	631	1.4*	466	-	-	1.5	364	1.6‡	360	1.6‡	356	1.5	375	1.6	375	1.6	369
	2	-	-	-	-	-	-	1.4	442	1.5	383	1.5	385	1.5	386	1.5	328	1.5	379
550	1/2	1.6	382	1.6	370	-	-	1.6	346	1.7‡	341	-	-	1.6	356	1.7‡	351	-	-
	1	1.3‡*	688	1.4‡*	450	-	-	1.6	358	1.7‡	341	-	-	1.6	367	1.7‡	348	-	-
	2	1.1*	794	-	-	-	-	1.4	472	1.6	362	1.6*‡	358	1.5	374	1.6‡	357	1.6‡	349

[a] Unloaded tray. [b] Puff index. [c] Hardness, m-g, torque. * Bed not fluidized. ‡ Burned or overtoasted.

TABLE 2.3--Effect of process variables on product characteristics - white wheat bulgur

Air temp. °F	Tray loading, lb/ft^2	Air velocity, f.p.m.[a] 965 Residence time, seconds 15 PI[b]	15 H[c]	25 PI	25 H	35 PI	35 H	1650 Residence time, seconds 15 PI	15 H	25 PI	25 H	35 PI	35 H	2200 Residence time, seconds 15 PI	15 H	25 PI	25 H	35 PI	35 H
350	1/2	1.4	426	1.5	380	1.5	368	1.5	377	1.5	363	1.5	371	1.5	383	1.5	381	1.5	370
	1	1.3*	588	1.3*	446	1.5*	405	1.5	396	1.5	374	1.5	372	1.5	387	1.5	386	1.5	385
	2	-	-	-	-	-	-	1.4	480	1.5	391	1.5	391	1.3	461	1.5	393	1.5	386
500	1/2	1.5*	377	1.6	371	1.6*‡	364	1.6	367	1.6‡	371	-	-	1.6	363	1.6‡	364	-	-
	1	1.3*	631*	1.4	466	-	-	1.5	364	1.6‡	360	1.6‡	356	1.5	375	1.6	375	1.6	369
	2	-	-	-	-	-	-	1.4	442	1.5	383	1.5	385	1.5	386	1.5	378	1.5	379
550	1/2	1.6	387	1.6*‡	-	-	-	1.6	346	1.7‡	341	-	-	1.6	356	1.7‡	351	-	-
	1	1.3*‡	688	1.4*‡	-	-	-	1.6	358	1.7‡	341	-	-	1.6	367	1.7‡	348	-	-
	2	1.1*	-	-	-	-	-	1.4	472	1.6	362	1.6*‡	358	1.5	374	1.6‡	357	1.6‡	349

[a] Unloaded tray. [b] Puff index. [c] Hardness, m-g torque. * Bed not fluidized. ‡ Burned or overtoasted.

This information will enable us to operate the Surface Combustion Puffer to obtain optimum conditions for puffing, texture, and toasting on a variety of bulgurs made by different processes and to determine differences brought about by changes in processing.

One aspect of textural measurement, not presently understood, shows up in these two series. At comparable puff index, the puffed white bulgur samples consistently have textural values approximately 100 M-gm higher than the puffed red bulgurs. In other samples, differences of this magnitude are usually easy to detect in bite tests. Detection was difficult with these samples. This discrepancy has been noted before.

The hardness tester is essentially a burr mill connected to a dynamometer to measure the force required to grind the sample. Spacing between the burr is adjustable. Standard operation is with the burrs close together so that the puffed bulgur is first broken into small particles in the top of the equipment, then it goes between the burrs where the main grinding force is exerted. We are thus measuring the hardness mainly of the endosperm portion of the sample and perhaps not the total bite characteristics. To test the effect of changing the burr settings, a series of 5 puffed bulgurs were ground with burr settings from very fine to very coarse. The results were checked against subjective evaluations by a panel of 15 members. The present standard technique with the fine setting gave values that agreed best with panel judgments and gave the greatest spread of values (i.e., the greatest sensitivity).

While newer and more comprehensive methods of textural measurements need to be investigated, the standard technique currently in use appears sufficiently valid for present purposes.

SECTION 3

ADJUNCTS FOR USE WITH THE BULGUR WAFER

Adjuncts are a series of food items for use in small quantity with the basic shelter ration, the bulgur-type wheat wafer, only to provide variety and thereby relieve the monotony of a single-item diet. The adjuncts are not designed to provide particular nutrients, nor do they in any way attempt to alter the nutritional balance originally set for the ration.

We have formulated a number of dry mixes which can be prepared by addition of water and, in some cases, by heating to provide soups, sauces, gravies, spreads, toppings, etc. The development work on adjuncts has been substantially completed and reported in the annual reports for Fiscal Years 1962, 1963, and 1964. Some final developmental work is reported here on a one-package, cold-water, high-methoxyl pectin jelly and its evaluation in two Office of Civil Defense Shelter Experience Tests.

Cold-Water High-Methoxyl Pectin Jellies

The jellying ingredient for this product is prepared by drum drying a mixture of high-methoxyl pectin (Sunkist Growers #3430) and sugar dissolved in water at about 20-25% solids. The sugar/pectin ratio and pH of the jellying ingredient were varied, to observe the influence of this variation on the final jelly. A jellying ingredient with 15/1 sugar/pectin ratio adjusted to pH 6 was selected as best.

The basic recipe for the cold water, high-methoxyl, pectin jelly is:

	% by weight
Drum-dried sugar pectin mix (15:1 sugar/pectin, pH 6)	11.1
Sugar, granulated	87.1
Delta glucono lactone	1.8

Artificial flavorings and colors may be added in the quantities shown in Formulas for Adjuncts numbers 66 through 74 (Appendix, Report for FY 1964), to provide a variety of flavors.

Twelve cans of a dry mix of the cold-water jelly were prepared in two flavors, strawberry and raspberry, and furnished to Mr. George W. Wassmer, Staff Director, Material Technical Division, Material Office,

Office of Civil Defense, for trial in a shelter exercise in a Shelter Management Instructor Course at three locations throughout the country.

The ...as contained a plastic bag with pre-formed spout and a paper-cov...ed wire tape to serve in preparing and dispensing the jelly. The following instructions were provided on the label of each can:

> Cut top from can and remove plastic bag. Pour powder into bag by slipping top of bag over top of can and inverting. Fill can with water to mark on inside of can and pour water over powder. (Save can.) Close bag by twisting it about 4 inches above the mixture, and tie securely with wire tape; do not trap too much air. Mix contents by tipping the bag back and forth and kneading the contents for about 3 minutes. Place the jelly, bag and all, in the can and allow to stand until gelled (about 3 hours). To serve, cut off spout at bottom of bag and squeeze bag to extrude jelly.

Reports from two of the three Civil Defense courses where the jelly was used have been received. The comments were almost completely favorable. Users felt the jellies improved palatability of the ration. There were other comments such as raspberry flavor was more acceptable, strawberry too sweet, the jelly made them thirsty, and gel time should be shorter.

The gel time can be shortened only at the expense of a poorer texture. Three hours setting time appears practical since it allows a jelly to be prepared after one meal to be ready for the next. The jelly may be held satisfactorily for several hours; very little change in gel strength occurs in three hours.

One cogent comment on preparation was that the can opener issued in shelter supplies left jagged edges that might tear the mixing bags and possibly injure fingers. A change in instructions may be required. The plastic bag need not be placed in the can after mixing as called for in the instructions; this was merely a convenience. The bag may be placed on a table top just as well. The jagged edges should not interfere seriously with the removal of the plastic bag, pouring out of the can contents, measuring and pouring the water.

These tests provided other important information. First, the preparation of the jelly for serving was easy for users unfamiliar with the product. Second, it was prepared successfully with two different water supplies.

SECTION 4

STORAGE STABILITY

Storage stability of foods for stockpiling in shelters may well be the dominant factor in their true cost. In spite of our efforts, as well as those of many other workers in the field, to develop reliable, quick, objective measurement of shelf life, the one sure test remains long-term storage of the material at normal temperatures and periodic evaluation of various quality factors by subjective taste panel methods.

Contractural arrangements were made on June 29, 1962, with Oregon State University to conduct a five-year study of storage stability of the bulgur shelter wafer, and a similar study of 12 selected adjuncts was started one year later. Results emerging from these contract studies are discussed below.

Bulgur Wafers

The contract on wafer stability provides for taste panel evaluation of 16 types of bulgur wafers stored at three temperatures (40°, 70°, and 100°F). Several physical-chemical tests are also performed, to discover any correlations between changes in physical-chemical characteristics and panel evaluation. Such correlations, if they are found, might lead to quick, reliable, objective methods for surveillance testing of stockpiled wafers. The wafers are sampled at 6-month intervals.

Red and white wheats with appropriate protein content were provided by the Fisher Flouring Mills Co., Seattle, Washington. They processed part of each lot into bulgur by a pressure-cooking process. The remainder was processed into bulgur by atmospheric cooking at the Armeno Cereal Co., Westboro, Massachusetts. All bulgur was puffed by the Van Brode Milling Co., Inc., Clinton, Massachusetts, and made into wafers incorporating the following treatments:

Red Wheat		White Wheat
1	Pressure cooked, malt binder, nitrogen pack (REFERENCE)	9
2	Pressure cooked, malt binder, air pack	10
3	Pressure cooked, corn sirup binder, nitrogen pack	11
4	Pressure cooked, corn sirup binder, air pack	12

5	Cooked in atmosphere, malt binder, nitrogen pack	13
6	Cooked in atmosphere, malt binder, air pack	14
7	Cooked in atmosphere, corn sirup binder, nitrogen pack	15
8	Cooked in atmosphere, corn sirup binder, air pack	16

Taste-panel evaluation

One red-wheat and one white-wheat formulation, arbitrarily chosen, serve as reference samples and also serve as controls held at -18°F. The flavor of control samples is scored at each sampling period by means of a 9-point hedonic scale ranging from 1 for the lowest to 9 for the highest rating. Results of these judgments through 28 months of storage are given in Table 4.1. At any given sampling time, no significant difference was found between red wheat and white wheat wafers. No statement of significance is possible for the zero-time samples because they were not judged by the same panel. The variation in flavor scores as the test progresses appears to be only a variation in taste panel performance.

At each sampling period the reference formulations (numbers 1 and 9) stored at 40°, 70°, and 100°F are compared with their appropriate controls (stored at -18°) by means of a reference-preference test on a 9-point hedonic scale. Each of the other formulations is then compared with its appropriate reference sample by means of the same reference-preference test. Tables 4.2 and 4.3 compare mean scores of the 28-month samples with the original mean scores.

The analysis of variance for the 28-month storage data, within the three temperature groups and considering red wheat and white wheat separately, is shown in Table 4.4.

The protective effect of nitrogen packing on flavor becomes more pronounced as the storage test continues. The estimate of the economic value of inert gas pack previously reported (see Report "Bulgur Wafers and Adjuncts for Fallout Shelter Rations," 1964, p. 2) appears to be becoming more valid. Data in Tables 4.2 and 4.3 indicate that shelf life can be extended considerably by nitrogen packaging, particularly when associated with the protection imparted by malt sirup.

Whether malt sirup stabilizes the wafer by masking the rancid flavor or because it contains some particularly effective antioxidant is not clear. Its stabilizing effect, however, is becoming more pronounced as these storage tests continue. Its slight additional cost over corn sirup, one-third of a cent per pound of wafer, may well be justified.

Table 4.1

Flavor scores on a 9-point hedonic scale[a] of bulgur wheat wafers, formulations 1 and 9, stored at -18°F

	Storage time, months					
	0[b]	4	10	16	22	28
Red wheat	6.40	6.03	5.68	6.53	6.26	6.22
White wheat	5.72	6.41	5.82	6.42	6.20	6.39

[a] 1 is lowest, 9 highest.

[b] At 0 time, red wheat and white wheat samples were judged by different panels and, therefore, cannot be compared.

TABLE 4.2--Flavor scores of red wheat bulgur wafer, initially and after 28 months storage (reference-preference mean[a] scores)

Type of cooking	Packaging	Initial scores		Stored 28 months at:					
				40°		70°		100°	
		Malt[b]	Corn[b]	Malt	Corn	Malt	Corn	Malt	Corn
Pressure[c]	In nitrogen	5.25		5.31		5.36		5.03	
Pressure	In nitrogen	5.25	5.83	5.55	4.83	5.65	4.95	5.29	5.05
	In air	5.53	5.08	5.19	4.76	5.27	4.10	4.02	3.73
Atmospheric	In nitrogen	5.28	5.30	5.62	5.52	5.93	5.95	5.37	5.65
	In air	5.63	4.95	4.95	5.40	5.09	4.68	4.41	3.71

[a] 40 judgments.

[b] Wafer formulated with malt syrup or corn syrup.

[c] Reference samples compared with controls stored at -18°F.

TABLE 4.3.--Flavor scores of white wheat bulgur wafers, initially and after 28 months storage (reference-preference mean[a] scores)

Type of cooking	Packaging	Initial scores		Stored 28 months at:					
				40°		70°		100°	
		Malt[b]	Corn[b]	Malt	Corn	Malt	Corn	Malt	Corn
Pressure[c]	In nitrogen	5.28		5.16		5.05		5.19	
Pressure	In nitrogen	5.28	4.98	5.54	4.75	5.54	4.97	5.42	4.92
	In air	5.17	4.75	5.30	4.50	4.58	3.88	3.47	3.32
Atmospheric	In nitrogen	5.85	5.45	5.57	5.06	5.44	4.80	5.42	4.76
	In air	5.25	5.25	5.07	4.71	5.06	4.62	4.30	3.80

[a] 40 judgments.

[b] Wafer formulated with malt syrup or corn syrup.

[c] Reference samples.

TABLE 4.4.--Analysis of variance of flavor scores of wheat wafers

(28 months storage)

		40°F		70°F		100°F	
		Red wheat	White wheat	Red wheat	White wheat	Red wheat	White wheat
Source of Variation	DF	"F"	"F"	"F"	"F"	"F"	"F"
Cooking	1	5.25*	0.64	9.76**	3.06	2.99	5.14*
Packaging	1	5.69*	5.78*	38.56**	23.04**	84.22**	122.64**
Binder	1	2.38	20.71**	17.39**	18.65**	2.57	12.74**
Cook x pack	1	0.49	0.76	2.75	7.56	0.25	8.29**
Cook x binder	1	8.69**	1.43	7.42**	0.11	0.04	1.02
Pack x binder	1	2.68	0.00	2.76	0.01	3.02	1.03
Cook x binder x pack	1	0.26	0.27	0.01	0.34	2.44	0.14
Error mean square	245	1.36	1.40	1.53	1.54	1.87	1.35

(The single and double asterisks indicate statistical significance at the 5% and 1% points, respectively.)

Significant differences between the two cooking processes are beginning to appear. The panel showed a significant preference for atmospheric-processed bulgur over the samples made from pressure-processed bulgur. Since atmospheric-processed bulgur expands less during puffing than that processed under pressure, the stability difference may be due to the less porous structure in the atmospheric-processed material. Or it may be due to incipient degradation brought about by the higher process temperatures under pressure. Whatever the cause, we need to know more about the changes occurring in wheat during processing.

Chemical-physical determinations

At each sampling period, each lot of wafers is analyzed to determine percentage fat, peroxide number, thiobarbituric acid number, carbonyls, and diene values. Gas chromatograms (aromagrams) are also prepared. The contract was extended to include analysis of headspace gas for carbon dioxide, carbon monoxide, oxygen, and pentane.

Analyses have been completed on samples drawn from storage after 22 months. Changes are occurring in nearly all factors being studied. The changes are influenced to a varying degree by all the variables of interest except type of wheat. Temperature exerts the most consistent influence. Of particular interest are the changes in the so-called permanent gases or headspace gases (Table 4.5). Note that carbon monoxide has appeared only in the air-packed samples. Its appearance generally parallels the disappearance of oxygen. Carbon dioxide has appeared in all packs but generally to a greater extent in the air pack. The changes in these permanent gases in the headspace follow the same pattern as was reported in section I from the studies on the model system. Pentane, however, has not been observed in any of the wafer samples.

Analytical data are steadily accumulating. When sufficient changes in all chemical-physical factors and taste panel evaluations have taken place, correlations between the several chemical-physical values and panel results will be made. It is on the basis of these correlations that the value of any single analysis for predicting storage stability may be determined.

Adjuncts

The contract to determine the stability of adjuncts in normal storage provides for taste panel evaluation of twelve representative adjuncts: apple topping, beef soup, butterscotch topping, chili sauce, chocolate pudding, cream of chicken soup, curry sauce, Oriental sauce, paprika gravy, raspberry jelly, strawberry spread, wild cherry icing. Samples stored at 40°, 70°, and 100°F are to be compared with nitrogen-packed controls at -18°F. All sample adjuncts are packed in tin cans, with

TABLE 4.5.--Changes in oxygen, carbon dioxide, and carbon monoxide content of headspace gas above bulgur wafers after 22 months' storage (mean values)

Formulation	Packaging	Oxygen, %			Carbon dioxide, %			Carbon monoxide, area (mm^2) under peak		
		Storage temperature, °F								
		40°	70°	100°	40°	70°	100°	40°	70°	100°
1	Nitrogen	0.0	0.0	0.0	0.0	1.0	4.5	0.0	0.0	0.0
2	Air	9.5	6.25	2.0	1.0	2.0	12.5	1.0	124.3	242.7
3	Nitrogen	0.0	0.0	0.0	0.5	1.5	5.0	0.3	0.0	0.0
4	Air	12.25	4.5	2.0	1.0	2.0	8.0	1.0	119.0	201.3
5	Nitrogen	0.0	0.0	0.0	0.5	1.5	4.5	0.0	0.0	0.0
6	Air	13.35	5.0	2.0	1.0	1.0	8.0	1.0	48.0	162.8
7	Nitrogen	0.0	0.0	0.0	0.5	2.0	1.0	0.0	0.0	0.0
8	Air	19.35	10.5	2.0	0.5	2.5	2.0	1.0	99.8	159.3
9	Nitrogen	0.0	0.0	0.0	0.5	2.0	2.0	0.0	0.0	0.0
10	Air	21.85	7.5	2.0	0.5	2.5	4.0	1.0	120.8	192.3
11	Nitrogen	0.0	0.0	0.0	1.0	2.0	4.0	0.0	0.0	0.0
12	Air	23.0	7.5	2.0	1.0	2.0	8.0	38.5	98.5	192.3
13	Nitrogen	0.0	0.0	0.0	1.0	2.0	4.0	0.0	0.0	0.0
14	Air	24.5	8.0	2.0	1.0	4.0	8.0	31.5	105.0	201.0
15	Nitrogen	0.0	0.0	0.0	1.0	2.0	4.0	0.0	0.0	0.0
16	Air	21.0	5.2	2.0	1.0	3.25	8.0	31.5	98.0	195.8

nitrogen atmosphere in half the samples and air in the other half. Five adjuncts low in moisture are packed without desiccants; the other seven higher in moisture are packed with and without in-package desiccant. Table 4.6 lists the package and storage treatment given all 12 adjuncts. They are sampled at 6-month intervals over a period of 5 years.

Taste-panel evaluation

At the beginning of the storage study and at each test period all samples were evaluated before, during, and after preparation, by an expert panel of four judges. Odor, texture, color, and ease of preparation are scored on a 6-point hedonic scale (0-normal to 5-extremely off). The prepared adjuncts were evaluated each time for flavor by a large taste panel using a 9-point hedonic scale.

After 18 months' storage the expert panel gave only slightly altered scores on preparation. In most cases the difference lay in the color of the dry mix or the rehydrated mix or both stored at the higher temperatures. Only the cream of chicken soup mix exhibited a change of any real consequence; the color score went from 0 to 3 at all three steps in preparation for the two samples stored at 100°F. Some samples exhibited slight changes of texture of the rehydrated sample, but only one, the Oriental sauce, showed a major change: it became too thick for convenient dispensing.

Large-panel evaluations of flavor have been completed on all samples after 18 months' storage. Mean flavor scores are given in Table 4.7.

The protective action of nitrogen packaging is shown particularly by the flavor scores on apple topping, Oriental sauce, and paprika gravy. All adjuncts, except beef soup, that contain desiccant scored higher, attesting to the protective effect of low moisture. The effects of high temperature are especially demonstrated by the lower flavor scores given apple topping, butterscotch topping, chicken soup, and Oriental sauce stored at 100°F. As storage continues, these trends will probably become more pronounced and the true value of the protective packing can be established.

Chemical-physical determinations

On the samples possessing flow characteristics, Bostwick Consistometer readings are made in order to have objective measurement of changes in consistency during storage. Samples in which consistency was noted as altered by the panel of expert judges were also observed to be changing in Consistometer readings. The Oriental sauce, which received a subjective score of 3 at the end of 12 months, was too thick to give a Consistometer reading.

Table 4.6

Storage plan for each adjunct under test

Code number	Treatment	Can code
1[a]	-18°F, nitrogen, desiccant (for products packed with desiccant)	1-N-D
1[a]	-18°F, nitrogen (for products packed without desiccant)	1-N
2[b]	40°F, nitrogen	2-N
3[b]	70°F, nitrogen	3-N
4[b]	100°F, nitrogen	4-N
5[b]	40°F, oxygen	2-O
6[b]	70°F, oxygen	3-O
7[b]	100°F, oxygen	4-O
8	40°F, nitrogen, desiccant	2-N-D
9	70°F, nitrogen, desiccant	3-N-D
10	100°F, nitrogen, desiccant	4-N-D
11	40°F, oxygen, desiccant	2-O-D
12	70°F, oxygen, desiccant	3-O-D
13	100°F, oxygen, desiccant	4-O-D

[a] Control samples.

[b] Five low-moisture adjuncts: apple topping, butterscotch topping, raspberry jelly, strawberry spread, and wild cherry icing, packed only without desiccant and subject to only this portion of test.

TABLE 4.7.--Flavor scores of all adjuncts after 18 months' storage

Code number[a]		Adjunct[b]											
		AT	BFS	BT	CHS	CHP	CCS	CuS	OS	PG	RJ	SS	WCI
1 1-N	-18°	6.51	--	5.69	--	--	--	--	--	--	5.35	5.78	5.94
1 1-N-D	-18°	--	5.25	--	4.89	6.12	5.83	4.81	5.79	5.76	--	--	--
2 2N	40°	6.10	5.10	6.14	4.70	5.85	5.51	4.88	5.80	5.80	5.87	5.67	6.22
3 3N	70°	6.15	5.53	6.25	4.72	5.85	5.21	4.92	5.95	5.95	5.86	5.84	6.42
4 4N	100°	6.61	--[c]	6.04	4.42	5.77	4.99	4.92	5.06	5.06	6.44	6.41	6.49
5 2-0	40°	6.63	6.25	6.50	4.56	5.59	5.52	4.58	4.94	4.94	5.76	6.36	6.42
6 3-0	70°	6.29	5.47	6.40	4.63	5.97	5.14	4.85	5.39	5.39	6.55	6.39	6.57
7 4-0	100°	4.97	--[c]	5.80	4.44	5.75	4.32	4.29	4.65	4.65	5.27	6.32	6.57
8 2-N-D	40°	--	5.47	--	5.33	6.06	5.81	5.92	5.64	5.64	--	--	--
9 3-N-D	70°	--	5.42	--	4.98	6.10	6.01	5.21	5.93	5.93	--	--	--
10 4-N-D	100°	--	5.22	--	5.01	6.17	5.84	5.48	5.54	5.54	--	--	--
11 2-0-D	40°	--	5.57	--	5.21	5.92	6.10	5.42	5.30	5.30	--	--	--
12 3-0-D	70°	--	5.11	--	4.31	5.97	5.02	5.48	5.78	5.78	--	--	--
13 4-0-D	100°	--	5.30	--	4.90	5.86	5.30	5.52	5.85	5.85	--	--	--
LSD (.05)		0.47	0.86	0.50	0.56	NSD	0.27	0.51	0.55	0.50	0.55	0.44	0.43

[a] See Table 4.6 for storage conditions.

[b] AT - apple topping, BFS - beef soup, BT - butterscotch topping, CHS - chili sauce, CHP - chocolate pudding, CCS - cream of chicken soup, CuS - curry sauce, OS - Oriental sauce, PG - paprika gravy, RS - raspberry jelly, SS - strawberry spread, WCT - wild cherry icing.

[c] Withdrawn from testing after 6 months because of hard swells.

The headspace gas of all samples is analyzed for carbon dioxide, and fhe nitrogen-packed samples for oxygen. If appreciable oxygen is detected in nitrogen-packs, the samples are rejected and new ones drawn. Carbon dioxide is considered an objective measure of degradation.

Carbon dioxide first appeared in headspace gas of a few samples after they had been stored for six months at high temperatures. In fact, both samples of beef soup stored at 100°F were hard-swells with very high carbon dioxide content at 6 months; they had to be removed from the test. Carbon dioxide has continued to increase in all samples stored at 100° with no desiccant, up to the 18-month check. Air-packed samples developed consistently more carbon dioxide than did nitrogen packs. No samples packed with desiccant have shown any significant quantity of carbon dioxide up to 18 months.

Examination of the components of the various adjuncts suggests that a Maillard-type browning reaction may be responsible for the development of carbon dioxide, since only the adjuncts containing no amino acids, peptides, or proteins are still free of this compound in the headspace gas.

Correlation of the results of these objective tests with the subjective results from panel evaluations will be made when enough data have accumulated. The value of these tests for surveillance sampling will rest on these correlations.

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13. ABSTRACT

Vapors from rancidifying bulgur and from autoxidized methyl linoleate, a model compound, are being analyzed and identified by gas liquid chromatography and mass spectrometry. Thirty-one compounds have now been tentatively identified in studies on the model system. The presence of some of these compounds in vapors from rancid bulgur has been verified. We have made further study of gun-puffing and hot-air puff-drying as alternate means of preparing wheat ingredients for the wafer. Development of a jelly that sets with cold water essentially concludes development work on adjuncts.

Long-term (five-year) storage studies of bulgur wafers and adjuncts are continuing. Taste panel evaluations to date indicate that shelf life of both types of products can be extended with nitrogen-packaging. The use of malt instead of corn syrup in wafer formulation and of in-package desiccants with adjuncts also extends shelf life. Chemical-physical measurements of changes taking place are being made on duplicates of the samples evaluated by the taste panel to find a test that will correlate with organoleptic evaluations, but as yet no meaningful correlations have been found.

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